LabNotes Fall 2002

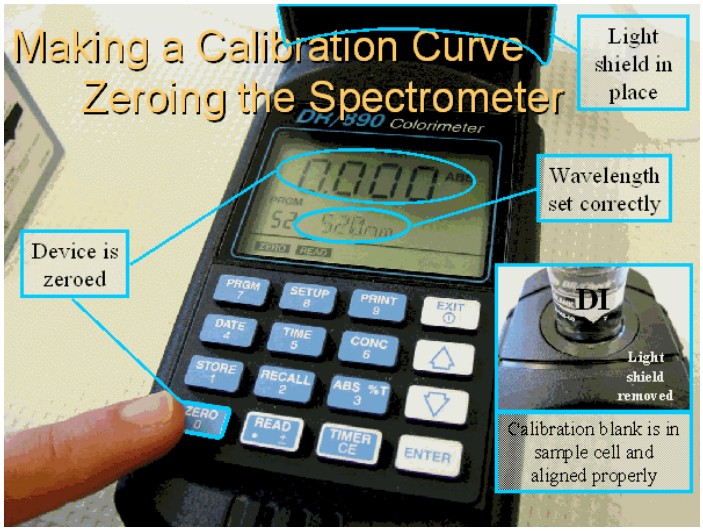
New Training Initiative Launched

Web-based, multimedia training is now available on the Laboratory Certification and Registration Program web site. The first training program using this new approach covers total residual chlorine (TRC) testing. A series of additional tests will be covered in the future as the program builds a training library for lab analysts.

In other training developments, audit chemist, Richard Mealy has been named Laboratory Training Coordinator and a new training page has been added to the Lab Cert. Program web site.

TRC Using DPD in Vacuum Ampoules

The Lab Cert. Program will place additional emphasis on laboratory training in the coming years, using both traditional face-to-face training and computer technology. Web-based training has the advantage of eliminating travel time and overnight stays, and allows the analyst to learn at his or her own pace.



A slide demonstrating a procedure in the Total Residual Chlorine training PowerPoint presentation available on the training page of the web site.

The TRC training focuses on the DPD (N,N-diethyl-p-phenylenediamine) method using vacuum ampoules. The training program consists of a Microsoft PowerPoint presentation that covers

Continued on next page

Also in this Issue:

Contact us at: (608) 267-7633 or LabCert@dnr.state.wi.us
Web Site: www.dnr.state.wi.us/org/es/science/lc

***Training,** continued.*

the TRC test in detail, supported by a video that demonstrates the preparation of a calibration curve. This represents a new approach to delivering training for the Lab Cert. Program and is the first in a planned series of training programs that will cover a range of analytes and analytical techniques. Lab Cert. summer intern, Micah Berman prepared the presentation and video while on break from UW-Madison where he studies medicine and music.

The web site offers links to download a stand-alone PowerPoint viewer in Mac and PC formats, so the presentation can be viewed without the full-blown software package. The video is offered in several common formats to meet the needs of most labs. If the lack of an Internet connection or slow download speeds prevents access, the entire TRC training package is also available on compact disk (CD). Contact the Lab Cert. Program at (608) 267-7633 to order a free TRC CD for mail delivery.

A Training Page

Recent visitors to the Lab Cert. web site will likely have noticed that the new TRC training and all the existing laboratory training programs have been organized under a new training web page. Formerly, training materials were found on the "Lab Toolbox" web page. A "Training" button has been added to the Lab Cert. home page linking visitors to the new page. The goal of the new page is to present an organized and accessible one-stop shop for laboratory training.

The Lab Cert. Program will be working closely with the Certification Standards Review Council, the WSLH and the laboratory community to select future topics to expand this training library.

New Training Coordinator

Many lab analysts will be familiar with Rick Mealy because he has presented a variety of training courses over the past four years. Rick was recently named Laboratory Training Coordinator, in addition to his existing duties as an audit chemist, to serve as the program's point person for training issues. He will work closely with the Wisconsin State Laboratory of Hygiene and other stakeholders to pursue the shared goal

*See **Training** on page14*

LabNotes

Newsletter of the Laboratory Certification Program

LabNotes is published twice annually by the Wisconsin DNR Laboratory Certification and Registration Program. For information about distribution or to make suggestions for future articles, contact the editor.

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LabNews

National Environmental Methods Index (NEMI)

The EPA and U.S. Geological Survey now offer a free web-based clearinghouse of environmental monitoring methods. The index provides method summaries of laboratory and field protocols for water quality analyses. The system provides a way to compare methods to find the method that best meets the users monitoring needs. □

www.nemi.gov

Standard Methods 20th Ed. Approved

The EPA has approved the 20th edition of Standard Methods for the Examination of Water and Wastewater. While most methods in the 20th edition are now approved for regulatory compliance, facilities and laboratories must still check the relevant Wisconsin Administrative Codes for methods approved for Wisconsin compliance programs. (See related article on page 13.). □

Operator Certification Exams

DNR will hold Wastewater, Drinking Water and Septage Operator Certification exams on May 7, 2003 in DNR Regions around the state. Check the Op Cert. web site for details, as they become available. Application packets will be mailed in February 2003. □

www.dnr.state.wi.us/org/es/science/opcert

2003 Conferences, Meetings

MWAA Winter EXPO

The Midwest Water Analysts Association has scheduled Winter EXPO 2003 for January 31 at the Bratstop Banquet Center in Kenosha. Contact Larry Dressel at (630) 369-5586 for info.

Government Affairs Seminar

The Government Affairs Seminar will be held February 27, 2003 at the Marriott Madison West, in Madison.

WRWA Annual Conference

The Wisconsin Rural Water Association holds its annual conference on March 26 through 28, 2003 at the Green Bay Regency Suites and KI Convention Center complex. Call (715) 344-7778 or visit their web site for more information.

www.wrwa.org

WWA Annual Conference

The Wisconsin Water Association (formerly AWWA WS) annual conference is scheduled for September 24 through 26, 2003 in Middleton. Contact Jack Albrechtson at (608) 831-6554 for more information.

WWOA Annual Conference

The Wisconsin Wastewater Operators Association annual conference is to be held October 20 through 24, 2003 in the Wisconsin Dells. Check the WWOA web site for more details. □

www.wwoa.org

WRWA Training for Lab Analysts

The Wisconsin Rural Water Association offers Lab QA/QC Training and a Hach Class free of charge. The classes are open to non-members and no registration is required.

Lab QA/QC Training

Date	Location
January 23, 2003	Prairie du Chein Huckleberry's
February 20, 2003	Monroe
March 6, 2003	Winneconne
April 3, 2003	Ashland

Hach Class

Date	Location
April 17, 2003	Rhinelander
May 15, 2003	Plover
May 29, 2003	Eau Claire, Holiday Inn

Contact Rural Water at (715) 344-7778 or visit their web site for more information. □

www.wrwa.org

Tech. College Training Offered

Lab Analysis 1 – April 1-2, 2003

Offered by Blackhawk Technical College, Janesville. Call Moraine Park Technical College at (800) 221-6430 to register. □

Program Administration

NR 149 Revision Update

The last meeting of 2002 of the NR 149 Revision Advisory Committee (RAC) was held November 12. To date, the RAC has considered changes in three major areas: certification and registration structure, proficiency testing, and the application and renewal process. The agreements made by the RAC will constitute the basis for the first draft of the new Laboratory Certification and Registration Rule.

Certification and Registration Structure

The RAC has recommended establishing three matrix types for granting certification and registration: drinking water, non-drinking water aqueous, and solids. "Non-drinking water aqueous" would include domestic and industrial wastewaters, groundwater, leachates, and TCLP extracts. The "solids" matrix would encompass soils, sediments, solid wastes, materials for hazardous waste characterization, and non-aqueous liquids. The "drinking water" matrix would be reserved for samples regulated under ch. NR 809, Wis. Adm. Code and the Safe Drinking Water Act (SDWA). Wastewater sludges (biosolids) may fall into the "non-drinking water aqueous" or the "solids" category depending on the amount of settleable solids present in a sample.

The increased clarity provided by the proposed structure should prove beneficial for data users and those who subcontract analyses.

The RAC agreed to offer certifications and registrations following a three-tiered structure. The structure endorsed by the RAC uses matrix/technology or method/analyte or analyte group as the unit of certification and registration. For the drinking water matrix, the second tier would be method. For all other matrices, the second tier would be technology. Technologies would include colorimetric, gravimetric, electrometric, and titrimetric techniques, as well as flame and graphite furnace atomic absorption, inductively coupled plasma, gas chromatography, gas chromatography/mass spectrometry, and high performance liquid chromatography.

The default option for the third certification and registration tier will be analyte. However, the Department will define "analyte groups" for appropriate compound classes, such as volatile organic compounds (VOCs), semivolatiles, and PCBs. Some of our existing groupings, such as nitrogen pesticides, will no longer be offered and for these classes of compounds laboratories will be required to seek certification analyte by analyte. Drinking water analyte groups will be those mandated by the SDWA, which at the present time include VOCs and haloacetic acids.

Although the certification and registration structure recommended by the RAC appears to be more complex than what the Department offers currently, the new structure should be a more faithful indicator of a laboratory's analytical capability. For instance, a laboratory now certified to perform ammonia could be analyzing the compound electrometrically (probe), by colorimetry, or by titration. The proposed structure eliminates guessing the analytical technique used since the laboratory would be certified or registered for each individual technique it chooses to perform, for example, non-drinking water aqueous/ electrometric/ammonia.

The increased clarity provided by the proposed structure should prove beneficial for data users and those who subcontract analyses, particularly in cases where the technique is specified by a permit or where achieving a specific detection limit is tied to the choice of analytical technique.

Proficiency Testing

The RAC offered several recommendations to improve administering requirements for proficiency testing (PT) samples or reference samples. To renew their certification or registration, laboratories are currently required to analyze a set of PTs successfully during a limited period within the certification and registration year. The RAC agreed it would be beneficial to expand this window by allowing laboratories to analyze PTs for renewal any time during the certification and registration period. Results for renewal would have to be submitted to the Department by August 15th. This should

minimize having to analyze "immediate response" PTs to renew a certification or registration.

Instead of mandating analysis of specific PTs in the Laboratory and Certification and Registration Code, the RAC recommended that the Department publish a list of required PTs periodically, after obtaining concurrence from the Certification Standards Review Council. This mechanism should enable the program to consider availability and suitability before proposing the analysis of a PT while at the same time giving the program the ability to address emerging needs without engaging in a rule revision.

As is currently the case, laboratories that fail to submit acceptable PTs before the August 15th deadline will not be eligible for renewal of their certifications or registrations. The RAC has discussed some of the provisions that currently tie enforcement to PT failures. The RAC has made some preliminary recommendations in this area and they will be considered more fully at future meetings.

Application and Renewal

The RAC felt that devising an annual renewal form would greatly improve the current procedures for renewing certification and registrations. The form would enable laboratories to update reference methods and contact information, or drop technologies, methods, or analytes for which certification and registration was no longer needed. The Department in turn would have more accurate information about the laboratories in the Certification and Registration Program.

The RAC discussed different types of applications (initial, revised, transfer of ownership, and reciprocity) at its most recent meeting as well as conditions for reinstatement after enforcement and non-renewal. The RAC also identified information to be included with the different types of applications.

Additional Information

Meeting agenda, notes and most handouts are available on the NR 149 Revision web site. A list of RAC members is on page 14 of this newsletter. Contact your representative on the RAC or Diane Drinkman, RAC Leader at diane.drinkman@dnr.state.wi.us if you have questions or if you wish to be added to electronic distribution lists. The next RAC meeting will take place on January 14, 2003. □

www.dnr.state.wi.us/org/es/science/lc/nr149

LC Program Taking Nominations for 2003 Registered Lab of the Year

The laboratory certification program is accepting nominations for the 2003 Registered Lab of the Year Awards. The awards recognize outstanding registered laboratories for their commitment to producing data of exceptional quality, and will be presented to their recipients before the Natural Resources Board this coming March.

Awards are presented in two categories: Small Facility (wastewater treatment plant labs with flows less than 1 mgd, or labs that perform a limited array of tests), and Large Facility (wastewater treatment labs with flows greater than 1 mgd, or labs that perform a wider array of more complex tests).

Nominees for Lab of the Year must be registered facilities located in Wisconsin. Certified laboratories will not be considered. Anyone can nominate a lab for one of the awards (you don't have to be a DNR employee), but labs may not nominate themselves. There is no limit on the number of times that a lab may be nominated, and labs may be nominated for (or presented) an award in consecutive years. A nomination committee will decide award winners, and the awards will be presented to the winners at the March 2002 meeting of the Natural Resources Board at DNR's central office in Madison.

To nominate a laboratory for 2002 Lab of the Year, contact Greg Pils at (608) 267-9564 or gregory.pils@.dnr.state.wi.us for a nomination form. Completed nomination forms must be received by January 24, 2002. A nomination form can be found at the end of this newsletter. □

Quality Matters

By Donalea Dinsmore, DNR QA Coordinator

Quality Forum - Chemical Names

Even though we may speak the same language, ambiguous meanings and miscommunications are common. Many formal chemical names are cumbersome and have multiple synonyms. Industry groups use trade names or shorthand designations for chemical products. Members of the public (interest groups) interested in chemical contaminants in the environment may not make necessary connections between the multiple chemical names. A room full of chemists might be able to draw the structure of 1,1'-(2,2,2-trichloroethylidene)bis(4-chloro)benzene, a CAS name, but may not understand its relevance to the environment until it is called by one of its common names, p,p'-dichlorodiphenyltrichloroethane or DDT. A common language would help.

EPA developed data standards and associated business rules to establish a common language for identifying chemicals in regulations, databases, publications and on websites. In addition to the CAS number and CAS name, the data standard requires EPA to establish an EPA Registry Name and, for substances that do not have CAS numbers, EPA will provide an EPA Identifier number. Once implemented, the data standards will facilitate electronic data transfer from states or industry to EPA. Chemicals will be named consistently in regulations across EPA programs. Data from states to EPA will use this standard. Eventually, electronic data submittals to DNR for compliance programs may rely on this data standard rather than the STORET numbering system that EPA no longer maintains.

EPA's Office of Environmental Information (OEI) has developed a system of registries to support the data standards program and numerous information technology initiatives including data exchange. Registries for substances (chemicals, biological organisms, and physical properties) are searchable databases accessible from EPA's website. The chemical registry system (CRS) includes not only the required elements of the data standard but also lists synonyms, chemical formulas, and a table that identifies regulations in which the chemical appears. CRS and the substance registry system (SRS) are proving to be useful tools. These registries can be accessed on the EPA web site. □

www.epa.gov/crs www.epa.gov/srs

Draft Data Qualifier List Available

Over the last several months, members of the Quality Forum have developed a "standard" list of data qualifiers. These qualifiers are intended to maximize the data usability for multiple purposes. The goal is to communicate relevant information for data users to interpret the results. The list of qualifiers includes assumptions and any rules for use so the qualifiers can be used consistently. We hope that the various DNR compliance programs that accept laboratory data will adopt this list. If you are interested in obtaining a copy of the Qualifier List, please contact Donalea Dinsmore by e-mail at donalea.dinsmore@dnr.state.wi.us. □

Qualifying Data at the LOQ

Recently, DNR staff noticed that laboratories are using different conventions for qualifying results at the limit of quantitation (LOQ). Though at first it might seem that instances of analytical results falling exactly at the LOQ would be an uncommon occurrence, in fact it is relatively common. This raised concern because results with "J" flags are excluded from electronic compliance screens and calculations. Laboratory Certification rules indicate that results below the LOQ should be qualified (s. NR 149.15(3) Wis. Adm. Code) and rules for water compliance programs treat results at the LOQ as valid for compliance determinations.

When we investigated this concern, we identified two causes for qualifying data at the LOQ. We found that the BETWEEN function in some standard software systems (e.g. Oracle databases) includes values at both extremes of the range. Therefore, values equal to the LOQ were being inappropriately "J" flagged. In another case, results were qualified prior to rounding the results for significant figures.

We encourage laboratories to review their practices for qualifying data at the LOQ and update their reporting conventions as necessary. We recognize that it will take resources for labs to update their computer systems. In the interim, DNR will remove the "J" flag on results equal to the LOQ before loading the compliance data into the Groundwater Environmental Monitoring System (GEMS). □

Council Corner

By Paul Junio, Council Chair

A Happy Holiday season to all! Since last I wrote, the Council has filled two of its empty seats (no, we still have neither a solid and hazardous waste representative nor a farmer actively engaged in livestock production). Katie Edgington, from the City of Janesville Water Utility Laboratory will be representing Public Water Utilities, and Jim Kinscher, from Modine Manufacturing in Racine will be representing Industrial Laboratories. At this point, I'll use their words as a way of introduction:

Katie Edgington, Janesville Water Utility

I have been employed by the City of Janesville in the Water and Wastewater Utilities for almost ten years with the first seven of those years being in the laboratory. While working in the lab, I also administered the industrial pretreatment program. Currently, some of my responsibilities at the Water Utility include collecting water at entry points to the distribution system, coordinating required analytical testing, reviewing results and reporting results to regulatory agencies.

I have a B.A. from Carthage College with a major in biology and a minor in chemistry and have attained certifications as a water and wastewater operator. My active memberships include AWWA, WEF and WWOA.

Katie Edgington

Jim Kinscher, Modine Manufacturing

I would like to take this opportunity to introduce myself. My name is Jim Kinscher and I am currently employed with Modine Manufacturing Company in Racine as Analytical Section Supervisor in the Chemical and Metallurgical Laboratory. Modine is an independent, worldwide leader in heat-transfer and heat-storage technology serving vehicular, industrial, commercial, building-HVAC (heating, ventilating, air-conditioning), and electronic markets. Modine develops, manufactures, and markets heat exchangers and systems for use in various OEM applications. My primary responsibilities include database administration, overseeing the lab quality system to maintain NR149 and QS-9000 registrations, supervision of all analytical testing (includes environmental, process control and production samples), and provide technical support to 16 United States production facilities. Before coming to Modine I spent four years with a commercial laboratory, Swanson Environmental, as a bench chemist and QC coordinator. Academically, I have a BS degree in chemistry from Marquette University.

I have known my predecessor, Dave Kollakowsky, for more than 10 years. His expertise in technical matters, understanding of quality systems and dedication and service to the council will be missed. Filling his shoes will be no small task, but I welcome the challenge. Serving as the industrial lab representative to the council is not only an honor but also a form of community service that will allow me to give something back.

Lastly, I want to take a moment to thank my employer for allowing me to pursue this position, Nora Erlandson, Industrial Pretreatment Coordinator with the Racine Wastewater Utility, for the nomination, and Department of Administration Secretary George Lightbourn for making the appointment that will allow me to serve.

Jim Kinscher

NR 149 Revision is Current Focus

As has been the case for much of the last year, the Council's activities have been focused on the re-write of NR149. While progress has been slow, it seems to me that we are better served to make things right by being patient, rather than rushing to judgement with a new Code. It is interesting seeing the ideas thrown around the room as we bounce ideas off of each other. Having totally opened the Code, I find particular interest in the "outside the box" notions that have been discussed (what would you think about NO PT SAMPLES?). If that teaser tweaks your interest level, feel free to join us at one of our meetings. The Lab Cert. web site has an entire section dedicated to the Committee proceedings, including Committee member contact information, meeting minutes, handouts and schedules. A contact list for RAC members is on page 14 of this newsletter. □

www.dnr.state.wi.us/org/es/science/lc/nr149

Proficiency Testing

Reference Samples for Renewal

The successful analysis of reference samples, also know as proficiency testing (PT) or performance evaluation (PE) samples, is one of only two explicit requirements for renewal of a laboratory's Wisconsin laboratory certification or registration. The only other requirement is the payment of the annual renewal fee. Laboratories must of course maintain a quality assurance system, follow approved methods, and otherwise comply with the requirements of ch. NR 149, Wis. Adm. Code, but successfully analyzing a reference sample for each test for which one is required is the keystone of maintaining certification or registration.

Isn't it surprising then, considering how critical reference sample analysis is, that this requirement can cause so much confusion? Here's a summary of the legal requirements and a timeline for the 2003 calendar year relevant to reference samples.

Requirements and Deadlines

Laboratories must analyze a reference sample for each test for which a reference sample is required between January 1 and August 31 each calendar year. The August 31 date is critical! Results must be reported to the Department prior to September 1 (meaning August 31 at midnight) or the laboratory's certification for the test will not be renewed on September 1. A lab that analyzes a reference sample on August 31 may not be able to get the acceptable result reported to the Department for renewal, unless the reference sample provider agrees to fax the graded results to the Department by midnight on August 31. Worse yet, a laboratory waiting until the last minute may get an unacceptable result, which means the laboratory's certification or registration will not be renewed for the test for the new certification or registration cycle.

A laboratory that is not renewed for one or more tests must immediately cease performing the analysis for the test(s), and subcontract the work out to a certified laboratory. If the laboratory wishes to have it's certification or registration reinstated for a test, an application, appropriate fees and all required background materials (including acceptable reference sample results) must be submitted to the Department. An onsite evaluation may be required prior to reinstatement.

DMR-QA

One major source of reference sample confusion involves the U.S. Environmental Protection Agency's Discharge Monitoring Report – Quality Assurance (DMR-QA) studies. This EPA requirement is separate and distinct from Wisconsin Laboratory Certification and Registration requirements. Laboratories may attempt to participate in a water pollution proficiency testing study that meets both the EPA DMR-QA requirements and the Wisconsin Laboratory Certification and Registration Program renewal requirements, but this involves complications and risks.

The complications are that the study provider must be both an EPA-approved (NIST) PT provider and a Wisconsin-approved reference sample provider. The study must open after the EPA deadline (usually some time in June, but not fixed for 2003) and the study results must be reported to the Department prior to midnight on August 31.

The risk is that laboratories participating in a summer study may not have adequate time to correct unacceptable results received on June or later studies. Providers typically take a month or more to grade study results, meaning that laboratories will not receive results from a June study until at the earliest July. If any results are unacceptable, there is little time to participate in a regularly scheduled study to get an acceptable result reported to the Department by August 31.

All laboratories are advised to participate in one or more PT studies early in the year to provide adequate time for study grading and data reporting before the August 31 deadline. Laboratories that also participate in the EPA DMR-QA studies should wait until EPA sends out the official notification for DMR-QA (the "308 letter" that includes all the forms for submittal) before analyzing any PT samples to meet the DMR-QA requirements.

Contact Phillip Spranger at (608) 267-7633 or by e-mail at phillip.spranger@dnr.state.wi.us for more information. □

Reference Sample Timeline for 2003

January 1	PT studies must close after January 1 to be counted for the 2003-2004 certification and registration cycle.
June	DMR-QA qualified studies must open after a date in June (as yet unspecified) set by the EPA.
August 31	Acceptable results must be received by the Department by midnight.
September 1	Laboratories that did not submit acceptable reference sample results for each test for which they are required prior to September 1 are not renewed for those tests, must cease performing analyses for the analytes, and are required to subcontract the work to a certified laboratory. Reapplication is necessary.

SDWA PTs Required by Method

Safe drinking water act certified laboratories are required to annually achieve acceptable results on PT samples for each analyte/analyte group and for each method used to report compliance monitoring results. Methods used solely for confirmation are excluded. To be certified for an analyte group (volatile organic compounds or haloacetic acids) laboratories must pass 80% of the individual analytes in the PT sample. The requirement to analyze PT samples by each method used is located in the EPA's "Manual for the Certification of Laboratories Analyzing Drinking Water," March 1997.

The Wisconsin Laboratory Certification and Registration Program rule incorporates the EPA Drinking Water Certification Manual by reference (see s. NR 149.21, Wis. Adm. Code). In 1999, EPA promulgated the requirement for PTs by method in the Federal Register.

In Wisconsin, implementing the requirement for PTs by method is complicated by the fact that certification is offered by analyte and not by method. However, this does not exempt laboratories from meeting this requirement. The Wisconsin Laboratory Certification and Registration Program requires laboratories submitting applications for SDWA analytes to include PTs, MDL studies, and for organic analytes, IDC studies, for each method listed on the application.

The requirement to annually analyze PTs by each method used to report compliance results is checked during the regular onsite evaluations of drinking water laboratories. □

Total Trihalomethane PT Grading

The grading requirement for total trihalomethane (TTHM) PT samples has changed. Laboratories must now achieve acceptable results on all four individual trihalomethanes in a PT sample to pass. Failure on one or more analytes means the lab must analyze another PT sample and pass all four of the trihalomethanes in order to be certified to analyze compliance samples for TTHM.

Previously, laboratories that successfully analyzed a PT sample for three out of the four trihalomethanes, plus the correct total, could be certified for TTHMs. □

Drinking Water

Electronic Data Submittal System

The DNR Bureau of Drinking Water and Groundwater has developed a process for receiving monitoring data electronically for compliance with ch. NR 809, Wis. Adm. Code (public water supplies). The Kenosha County Division of Health is about to start submitting public drinking water microbiological data to this system. This will allow for more automated handling of laboratory data and rapid notification of unsafe samples. The DNR computer system notifies DNR staff of unsafe samples. Contact Ron Arneson at arnesr@dnr.state.wi.us or (608) 264-8949 if your laboratory is interested in providing data in this way. Check the Laboratory Services web page for more information. □

www.dnr.state.wi.us/org/es/science/ls/lab_data

Wastewater Focus

Wastewater Mercury Rule Now Final

Wastewater permits being issued for many dischargers will now contain requirements to monitor for mercury using new, sensitive test methods according to rules recently finalized by the DNR. Revisions to the Department's Mercury Strategy, in the works for almost two years, became effective on November 1, 2002.

Generally, the new rules:

- Formally promulgate, as an approved method in Wisconsin, Method 1631, *Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry*.
- Specify quality (also quantity) requirements, including sensitivity, for mercury data to be considered representative for regulatory purposes.
- Requires that permits reissued for the potentially most significant dischargers of mercury must require generation of representative mercury data.
- Provides a mechanism for the Department to grant alternative mercury effluent limitations (variances) that rely on pollutant minimization programs (PMPs) to demonstrate compliance. These alternative limits may be approved when numerical water quality limits would not be feasible for a discharger to achieve.

More specifically:

- The rules do not require use of method 1631 but only require that the method used be sensitive enough to quantify levels in the sample (down to a point). In fact, since municipal permittees will be required to monitor influent levels (typically containing greater than 20 ng/L and up to several hundred ng/L of mercury) in addition to effluent, labs may want to use a less-sensitive method or dilute the sample to obtain mercury concentrations appropriate for their equipment.
- The Department will require laboratories performing mercury analytical work for regulatory purposes to be specially certified under the emerging technology provisions of ch. NR 149, Wis. Adm. Code.
- Laboratories must report method blanks to their clients. Matrix spikes must meet certain criteria (71 to 125 % recovery).
- Sample collection methods must be consistent with Method 1669: *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels*. Note that this sampling method is performance-based and modifications may be made as long as quality requirements specified in the method and in DNR's rule are met.
- Permittees are required to report results of field blank analyses with each set of sample point data. Permittees must collect a field blank for each day samples are collected. Levels of mercury in field blanks must not exceed one-fifth the level in the sample (down to a point). If a field blank does not meet these maximum contamination criteria, the data will not be considered representative. The permittee should then take steps to eliminate sampling contamination prior to the next round of sampling.

The Department expects permittees to initially encounter some problems collecting uncontaminated samples as demonstrated by field blank results. Rather than not reporting sample point data when field blanks do not meet contamination criteria, permittees should report sample results, pointing out that field blank contamination is excessive and indicate what corrective steps they are taking.

Under no circumstances is a permittee allowed to report mercury data whose results have been reduced by the amount of field blank contamination.

The majority of the regulatory language that constitutes the revised Mercury Strategy is contained in ch. NR 106, Wis. Adm. Code. Approved methods for wastewater permit analyses are contained in ch. NR 219, Wis. Adm. Code. To find additional detail you may access the Wisconsin administrative rules on the Revisor of Statutes web site (see link below) From the index scroll down to "NR Codes".

www.legis.state.wi.us/rsb/code/codtoc.html

The Department is well aware of the difficulties associated with the generation of

quality, low-level data for mercury. We desire to work closely with permittees and labs and hope to provide sufficient training opportunities. Your suggestions on areas needing attention will be beneficial as we work through the problems.

The Department has a limited supply of 20-minute videos that demonstrate the "clean hands/dirty hands" sampling procedures. These videos will be provided free of charge upon request.

For additional information on the Mercury Rule, contact Tom Mugan in the Bureau of Watershed at (608) 266-7420 or at thomas.mugan@dnr.state.wi.us or DNR QA Coordinator, Donalea Dinsmore at (608) 266-8948 or at donalea.dinsmore@dnr.state.wi.us. □

BOD Procedure Clarified

BOD Holding Time is 48 Hrs.

This continues to be one of the questions most frequently asked of auditors. In addition to conflicting information between holding times specified in the reference method and the Code (ch. NR 219, Wis. Adm. Code), confusion remains regarding what date to use for the sample collection date. While the latter question would be easy to resolve if autosamplers were set to collect samples from midnight to midnight, that solution is not a practical one. Realistically, composite samples will be collected in part over each of two consecutive days.

The Department has established the sample collection date for composite samples as the day on which the majority of sample is collected. In a typical wastewater treatment plant, operators will remove samples from the autosampler at approximately 7:00 a.m. That means that only 7 hours worth of the sample was collected on the day the sample was retrieved, while 17 hours worth (over 70%) of the sample was obtained during the previous day. For DMR reporting purposes, the previous day's date should be recorded as the sample collection date. For determination of holding time, however, the date/time on which the sample was removed from the autosampler is used to mark the beginning of the allotted holding time period.

Standard Methods specifies that, "In no case start analysis more than 24 h after grab sample collection." Applicable state code (ch. NR 219. Wis. Adm. Code) and federal rules (40 CFR Part 136) states that the holding time is "48 hours". Note 4 of Table F in NR 219 does, however, state that, "For other composite samples, the holding time commences immediately after the samples are removed from the composite sampler. The time the sample spends in the sampler during collection does not count towards the maximum holding time." This information is not contained in the parallel table contained in 40 CFR Part 136. For Lab Cert. Program purposes, state law takes precedence over method requirements, and thus the enforceable holding time for BOD is 48 hours from the date/time the sample is removed from the autosampler.

Consequently, if a composite sample is retrieved from the autosampler at 7:00 a.m. on 12/25/2002, the facility has until 7:00 a.m. on 12/27/2002 to set the sample up and begin incubation for BOD. On its DMR, the facility should report the results of this sample on the line associated with 12/24/2002—along with flow data from that date—since most of the sample was actually collected on 12/24/2002.

BOD Sample Temp. 20 +/- 3°C

To clear up some confusion with inconsistent sample temperature references in the BOD method in *Standard Methods for the Examination of Water and Wastewater* (SM 5210B), the Laboratory Certification and Registration Program will evaluate labs audited for the BOD procedure based on a sample temperature of 20 +/- 3°C. Samples must still be incubated at 20 +/- 1°C, as required in the method.

The BOD method in the 20th edition of Standard Methods (5210B) requires that samples be brought to 20 +/- 1°C prior to making a dilution, but then directs the analyst to use dilution water that has been brought to 20 +/- 3°C. Further, the method requires that samples, which are stored at near freezing, be warmed to 20 +/- 3°C prior to analysis.

The 19th edition of Standard Methods uses a different set of sample temperature requirements, directing the analyst to warm samples to 20 +/- 3°C, bring samples to 20 +/- 1°C prior to making a dilution and to use dilution water at 20°C. The 18th ed. of Standard Methods uses yet another set of sample temperature requirements, generally

Continued on page 12.

***BOD Procedure Clarified**, continued.*

requiring samples and dilution water at 20°C (with no temperature range allowance).

Since all three of these editions of Standard Methods are allowed when analyzing Wisconsin compliance samples for BOD, it was necessary to "level the playing field" for labs and implement one consistent sample temperature, namely 20 +/- 3°C.

Prior to settling on 20 +/- 3°C for sample analysis and dilution water, the Program assessed how this sample temperature range (17°C to 23°C) might affect data quality. The consensus was that the effects are negligible. Also, the methods committee of Standard Methods had previously agreed to implement one standard sample temperature requirement (20 +/- 3°C) for the 20th edition, yet the change was not made (erroneously) in the final version. The draft of Standard Methods 21st ed. has been corrected to show 20 +/- 3°C and should be published in 2004. □

2,3-dinitrophenol Testing

The DNR Wastewater Program recently clarified that testing for 2,3-dinitrophenol is not required for compliance with Wisconsin Pollution Discharge Elimination System permits. Chapter NR 105, Wis. Adm. Code, does regulate dinitrophenols as a class of compounds, and testing may be required for 2,4-dinitrophenol and 2,5-dinitrophenol in some permits. A decision was made long ago to not require testing for the 2,3- isomer, but somehow the parameter found its way onto the WPDES permit application test list for organics in the acid extractable fraction for primary industries. Testing is also not required for the 2,6- or 3,4- or 3,5- isomers.

For more information contact Tom Mugan of the Watershed Bureau at (608) 266-7420 or at tom.mugan@dnr.state.wi.us. □

Cross Media Analytical Issues

Federal Changes to Mercury Method Affect Labs and Permitees

In the October 29, 2002 Federal Register, EPA promulgated Revision E to method 1631 (low-level mercury). In addition to revising the method, EPA added flexibility to modify clean sampling techniques, lengthened the allowable time between sample collection and preservation, and emphasized that plastic sample containers are inappropriate for these types of samples. Because these changes came after Wisconsin DNR finalized its administrative rules for regulating mercury in wastewater effluents, a number of laboratories have asked how EPA's rule affects the monitoring requirements in DNR's new rules.

When ch. NR 219, Wis. Adm. Code, was revised to incorporate additional low-level mercury methods, we overlooked Table F that addresses containers, preservation, and holding times. The table below illustrates the differences between NR 219 and the current federal register.

Some of the conflict between the EPA requirements and NR 219 is moderated by use of the emerging technology provision in ch. NR 149, Wis. Adm. Code, to recognize laboratories performing low-level mercury analyses. As part of that process, DNR verifies that each laboratory supplies either fluoropolymer (TeflonTM) or glass containers to clients and that the preservative is either bromine chloride or hydrochloric acid. High-density polyethylene (HDPE) containers have not been allowed. The October 29 Federal Register re-emphasizes that only fluoropolymer or glass are appropriate containers because comparison data shows increased mercury concentrations ranging from

Comparison of Wisconsin and EPA Low-Level Mercury Sample Handling Requirements

	NR 219	EPA Requirement
Container Types	Polyethylene, Glass	Fluorpolymer, Glass
Preservative	HNO3	BrCl or HCl
Time to preservation	Immediate	48 hours or 28 days (conditionally)
Holding Time	28 days	90 days
Method citations	1631D, 245.7	1631 E

15 to 240 percent in samples collected using HDPE containers.

Footnote 4 to Table F in NR 219 recognizes that samples may be held longer than the specified holding time if the permittee or laboratory has data demonstrating sample stability and if EPA issues a variance. The October 29 federal register indicates that stability data was submitted during the public comment period. As a result, revision E allows samples to be preserved within 48 hours or, if the sample is oxidized (digested) in the sample bottle, up to 28 days following sample collection. In addition, the method states that preserved samples are stable for 90 days. Based on the information presented in the Federal Register, DNR believes that it is unnecessary to request a variance from EPA. DNR will recognize the federal regulations for mercury preservation and holding time until the preservation table in NR 219 is revised again.

For additional information about the new DNR rules, see the article entitled *Wastewater Mercury Rules Now Final* on page 10 of this newsletter. If you have questions about requirements for low-level mercury analyses, contact Donalea Dinsmore by e-mail at donalea.dinsmore@dnr.state.wi.us or Tom Mugan at thomas.mugan@dnr.state.wi.us. □

Which Edition of Standard Methods Must I Use?

There has always been confusion over which edition of Standard Methods is required for analysis:

- Chapter NR 219, Wis. Adm. Code, specifies the analytical test methods and procedures to be used for WPDES permit compliance, and the 18th edition is cited here.
- The Laboratory Certification and Registration Code (ch. NR 149, Wis. Adm. Code) lists the 19th edition as an "authoritative" source.
- The 20th edition has been out for several years now, and has been recently "approved" by the EPA.
- The 21st edition is due out in 2004.

Just when you thought the situation was significantly "muddied", the EPA stirred the waters once more with the release of the Federal Register for Wednesday, October 23, 2002. This Federal Register Notice contained a Final Rule revising wastewater and drinking water regulations to include updated versions of test procedures (analytical methods) for the determination of chemical, radiological, and microbiological pollutants and contaminants in wastewater and drinking water, with an effective date of November 22, 2002. For the four typical wastewater parameters (BOD, ammonia, total phosphorus, and TSS) as well as most other parameters, this Federal Register approves the procedures as written in each of the 18th, 19th, and 20th editions.

For a method such as that used for TSS (2540D), which has not been subject to editorial revision in over 15 years, citing any of the three editions makes no difference. On the other hand, the BOD procedure (5210B) has undergone significant revision in each of these three editions.

Another round of discussions with Department legal staff clarified that provisions in our Code allow us to accept any of the three editions as well. Consequently, laboratories will be allowed to cite Standard Methods procedures from any of the 18th, 19th, or 20th editions, so long as they are meeting all the requirements specified in the specific procedure they choose to reference. If, for example, a laboratory chooses to cite the 20th edition of Standard Methods as its reference method for BOD, then the facility must be able to demonstrate that all of the requirements specified in the 20th edition have been fulfilled. At this time, we can only state with certainty that there are differences between each of the three editions for BOD. We have not compared the other methods between editions for substantive changes. □

Verifying Sample Container Cleanliness

Sample contamination is an important consideration at nearly all levels of environmental testing, from sampling through final analysis. We all understand that it is essential to use clean glassware if one is to avoid contaminating samples during the course of an analysis, and recognize the importance of analyzing blanks in part as a check that glassware is free of contamination. And yet, it is not

uncommon for labs to pay little or no attention to one of the most common sources of sample contamination – contaminated sample containers. It is possible that this is due to the absence in administrative rule, authoritative source, or many methods of any mention of sample container cleanliness.

Certain methods do contain specific requirements for assuring sample bottle cleanliness (for example those for VOCs and trace metals) and where specified must be followed. However, even for those methods that do not, the Laboratory Certification Program strongly recommends that laboratories develop and implement a system for verifying that the sample containers they use or provide to clients are free of contamination.

There is no single "right way" to approach this issue. Various means by which the absence of contamination can be verified vary from analyzing "bottle" blanks from a percentage of sample containers at regular intervals, to retaining the certificates of analysis that are usually packed with new vendor certified "clean" containers. The bottom line: craft a strategy that best suits your operation. If you can ask yourself, "Would I feel comfortable defending the cleanliness of my lab's sample containers if challenged?" and answer "Yes" (Be honest with yourself!), you should be OK.

Feel free to contact the lab certification program or your auditor if you have any questions about verifying the cleanliness of your sample containers. □

***Training**, continued from page 2.*

of making high quality training available to the Wisconsin laboratory community.

As Laboratory Training Coordinator, Rick will pursue partnerships with other organizations that share the program's training mission. That will certainly include the WSLH, which has long played a central role in providing training to Wisconsin laboratories. But Rick will also work to widen the scope and reach of the training effort by partnering with other organizations, such as Wisconsin Wastewater Operators Association and Wisconsin Rural Water Association.

The first training product to result from the DNR/WSLH collaboration will cover metals by inductively coupled argon plasma (ICP or ICAP) emission spectrometry, and is due out in 2003. The Lab Cert. Program will also develop training materials related to e-coli and fecal coliform.

Training provides significant value to the Lab Cert. Program and to laboratories, sometimes doing more to improve data quality than formal certification. The success of the Program's training efforts to date is reflected in requests for Wisconsin laboratory training materials from five different countries and other states. Finding new, better and more efficient ways to deliver training, and coordinating our efforts with others should result in better service to Wisconsin's laboratory community and to data users.

Contact Rick Mealy at (608) 264-6006 or by e-mail at richard.mealy@dnr.state.wi.us for more information on laboratory training. □

NR 149 Revision Advisory Committee Membership List

Advisory Committee Member	Represented Constituency	Advisory Committee Member	Represented Constituency
George Bowman (608.224.6278) WSLH	State Laboratory of Hygiene	David Kollakowsky (414.221.2835) We-Energies	Industrial Laboratory
Debbie Cawley (920.432.4893) Green Bay Metro. Sewerage Dist.	Large Municipal Wastewater Plant	R.T. Krueger (715.478.2777) Northern Lakes Service	Wisconsin Environmental Laboratory Association
Joe Celmer (715.526.2181) Little Rapids- Shawano Mill	Paper Council	Marcia A. Kuehl (920.469.9113) MAKUEHL, Company	Demonstrated Interest in Laboratory Certification
Katie Edgington (608.755.3115) Janesville Water Utility	Public Water Utility	Ruth Klee Marx (715.842.7891) County of Marathon Health Dept.	Public Water Utility
Randy Herwig (608.592.3247) City of Lodi	Small Municipal Wastewater Plant	Steven Smith (608.224.2830 x 239) BT^2, Inc.	Non-Laboratory Data User
Paul Junio (920.261.1660) TestAmerica, Inc.- Watertown	Commercial Laboratory	Steven Sobek (608.267.3500) WI DATCP	Dept. of Agriculture, Trade and Consumer Protection
Jim Kinscher (414.636.1278) Modine Manufacturing	Industrial Laboratory	Randy Thater (262.524.3631) Waukesha Wastewater Plant	Municipal Environmental Group

2003 WISCONSIN REGISTERED LAB OF THE YEAR

NOMINATION FORM

The Wisconsin Department of Natural Resources Registered Lab of the Year Awards annually recognize registered laboratories for their outstanding commitment to producing high quality data. One award is presented in each of two categories: Small Facility and Large Facility. Small facilities include municipal wastewater treatment plant laboratories with a flow of less than 1 million gallons per day (mgd), or labs that perform limited types of testing (e.g., BOD, nitrogen, phosphorus, and solids). Large facilities may include major municipal wastewater treatment plant laboratories with flows greater than 1 mgd, labs that perform tests of greater complexity (e.g., oil and grease, metals, PCBs, VOCs) or labs that process a large volume of samples annually.

Nominees for the award must be registered facilities located in the State of Wisconsin. Certified laboratories will not be considered. Anyone, including DNR staff, can nominate a laboratory for one of the awards, but laboratories may not nominate themselves. There is no limit on the number of times that a laboratory may be nominated, and a laboratory may be nominated for (or receive) an award in consecutive years. In the event that insufficient nominations are received for either category, the Department reserves the right to not issue either award.

To nominate a registered laboratory for the 2003 Lab of the Year Award, complete the following form and include a summary of no more than three pages describing the reasons why you are nominating the laboratory for the award. Be sure to address the following considerations in your summary (Note – all considerations do not necessarily have to be addressed for a laboratory to be chosen to receive the award):

Nomination Considerations:

- Does the laboratory demonstrate a commitment to exceeding the minimum requirements for compliance with Department rules and guidance?
- Has the laboratory demonstrated a high level of commitment to correcting instances of non-compliance?
- What measures does the laboratory take to ensure the production of high-quality data?
- Does the laboratory's quality assurance program ensure that quality control data is used to evaluate and improve laboratory test procedures?
- For which other practices or achievements should the laboratory recognized?

Completed nomination forms must be received by January 24, 2002 in order for the candidate to be considered. A nomination committee will decide the Award winners. Please send the completed nomination form to: Lab of the Year Award, c/o Greg Pils – SS/BW, 101 S. Webster St., PO Box 7921, Madison, WI 53707-7921 or by FAX at (608) 266-5226.

Category: □ **Small Registered** □ **Large Registered**

Name of Laboratory Nominated: _____

Laboratory Director: _____

Laboratory Address: _____

Laboratory Phone #: _____

Nominator (your name): _____

Your Affiliation with Nominee: _____

Your Address: _____

Your Phone #: _____

LabNotes – Fall 2002

Volume 17, Issue 2
Wisconsin Department of Natural Resources

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